U.S. DEPARTMENT OF COMMERCE

TECHNICAL NEWS BULLETIN

OF THE

NATIONAL BUREAU OF STANDARDS

ISSUED MONTHLY

Washington, November 1934.—No. 211

CONTENTS

DEALERS' STOCKS OF CERTIFIED "ANALYTICAL" WEIGHTS

Because the Bureau had been quoted as opposed to the carrying of stocks of certified weights by dealers, a request for a recommendation on the subject was received from one of the large laboratory supply houses.

In brief, the situation is as follows: (1) The carrying of such stocks is very desirable if weights do not change appreciably from the certified values; (2) some sets of such weights do change so rapidly that certified values are of little use after the weights have been carried in stock even 6 months; (3) if weights change as rapidly as this, certification at the time of sale will give more accurately the values at that time, but these values may not be of great use to the purchaser, because such weights will continue to change after he receives them; (4) in general, with screw-knob weights, the constancy of each individual weight depends on the care and skill with which it is made rather than on the material of which it is composed; (5) when tested at this Bureau, some, and possibly most, of the least constant weights are eliminated by the present determination of variations with atmospheric humidity. Some sets, however, change seriously but slowly, and are not conspicuously variable with humidity.

In view of this situation, the only definite, unqualified recommendation that can be made is that weights be tested for constancy, 3 months being about the least time that is sure to give valuable data. Any individual set that has been so tested and found constant may safely be carried in stock.

STRENGTH AND ELASTICITY OF GROUND-COAT ENAMELS

In Technical News Bulletin No. 202 (February 1934), the method of determining the modulus of rupture of ground-coat enamels was described, and some indications of the partially completed experiments were discussed. This study of strength and elasticity

of some ground-coat enamels has been completed, and the following conclusions, applicable to the range of compositions studied, were reached:

1. Substitutions of sodium and boric oxides, one for the other, and of feldspar and flint, one for the other, caused no important change in Young's modulus of elasticity, although a definite trend was observed.

2. The changes in modulus of rupture caused by such substitutions were relatively small and are outweighed by other considerations.

3. The changes in modulus of rupture with composition, though small, were systematic. Substituting boric oxide for sodium oxide increased the strength, and increasing the flint to about 30 percent, with a corresponding reduction in feldspar below 30 percent, caused an appreciable decrease in strength.

4. The scatter in the results on modulus of rupture varied systematically with the composition, and varied in some cases directly and in others inversely with the strength.

5. The computed extensibility of the enamels varied within a range of 0.14 to 0.19 percent in direct proportion to the modulus of rupture.

6. The frequency distribution of the strengths of different specimens of the respective enamels was approximately symmetrical.

7. The stress-strain curves for these enamels were linear up to the stress causing failure, and their slopes were not influenced by the rate of loading.

8. The time factor had a marked effect upon the apparent strengths, failure occurring at substantially lower stresses with increased duration of the stress periods.

TALC IN WHITEWARE

An investigation is being conducted to determine some of the more important effects of MgO, introduced as a lime-free talc or as a lime-bearing amphibole (marketed as talc), in whiteware of the wall-tile type. The maximum percentage of talc (or amphibole) in the bodies is 43, the minimum 2.5. Each series of specimens was heated to 1,120, 1,180, 1,260, and 1,325° C (or, approximately, to cones 1, 6, 10, and 14).

The values obtained to date show that: (a) an appreciable variation of MgO content did not significantly alter the water absorptivity of the heated bodies, this property being influenced much more, relatively, by the CaO and by the alkalies in the feldspar; (b) the total linear shrinkage was decreased, for specimens heated to 1,260° C, from a maximum of 8 percent for a body containing no talc to a minimum of 0.7 percent for a body containing 43 percent talc. The shrinkage also was influenced significantly by the CaO in the amphibole and the alkalies in the feldspar; (c) moisture expansion was practically eliminated, but this must be accounted for in part by the low feldspar content; (d) the total linear thermal expansion to 700° C, considering specimens heated to 1,260 and 1,325° C only, was decreased from a maximum of 0.93 percent for a body containing neither alkalies nor MgO to 0.35 percent for a body containing 15 percent amphibole. In general the thermal expansions increased as the percentage of talc or of amphibole was increased over 30 percent and, other factors being equal, expansions of bodies containing talc were higher than of the comparable bodies of the amphibole series.

THE SYSTEM: K_2O-PbO-SiO_2

A preliminary survey of this system indicates that it contains three ternary compounds having the molecular ratios 1:2:2, 1:3:6, and 1:1:4, respectively. The first crystallizing as hexagonal plates, is uniaxial negative, has indices $\omega 1.93 \pm 0.01$, $\epsilon 1.72 \pm 0.01$, and the optic axis perpendicular to the cleavage. The second, crystallizing as well-defined fibers or lathes and resembling a short-fibered asbestos when crushed, has indices ranging from 1.69 ± 0.01 to 1.79 ± 0.01 extinction is parallel, and the optic axis is parallel with the longitudinal axis. The third crystallizes as rectangular platy granules of parallel extinction, and the indices range from 1.59 ± 0.005 to 1.65 ± 0.005. It is probably biaxial.

The linear thermal expansion to 300° C, and the softening temperature, of the three compositions, as glasses, are respectively: 0.46 percent and 395° C; 0.28 percent and 461° C; and 0.24 percent and 517° C.

EFFECTS OF WATER ABSORPTION ON HEATED CLAY BODIES

Several years ago an investigation was undertaken to determine primarily the effects of weathering and water absorption on the volume of several representative types of clay ware. The secondary consideration was an observation of glaze defects which might develop under these conditions in order to check the predictions resulting from

an autoclave treatment of similar specimens made at the beginning of the study.

After the autoclave treatment the remaining specimens were divided into two groups, one group being exposed to the weather and the other group being placed over water in a tight container. Over a period of 5 years, at 6-month intervals, the specimens were measured for length change and observation of the glaze coating.

In the preliminary autoclave treatment the specimens were held for an hour at a steam pressure of 150 lb/in.^{2} Two of the bodies, a semivitreous ivory and a cream-tinted wall tile body, showed no craze marks at all. After 5 years of exposure the semivitreous ivory specimen is still free from crazing and the cream wall tile body is showing a very few marks. The remaining bodies, a semivitreous white, a white, and a buff wall tile, and two terra-cotta bodies, showed more or less crazing in the autoclave test. All of these latter bodies on exposure began to develop craze marks within 6 to 12 months and to about the same degree as was apparent in the autoclave test. There was practically no change in the amount of crazing from then on to the end of the fifth year. There was little or no difference in the time or degrees of crazing developed on the specimens exposed to the weather as compared to those placed in the damp jar.

All of the specimens for both groups showed linear expansions of between 0.025 and 0.075 percent at the end of the first, and between 0.075 and 0.100 percent at the end of the second year. By the third year practically all the specimens showed increases of 0.1 percent or slightly more. No significant changes have occurred since. In general, there was a rather rapid increase during the first year, a very slight increase for the next 2 years, after which no further significant change appears to have taken place.

From the fact that both the crazing and expansions took place simultaneously and to the same degree in the case of both the groups exposed to the weather and those in the damp jar, it would seem that the changes are due primarily to water sorption.

COMPRESSION TESTS OF STRUCTURAL STEEL AT ELEVATED TEMPERATURES

In RP741 in the November number of the Journal of Research the strength in compression and the stress-strain relations are given for structural-steel shapes and for round bars at temperatures up to 945° C (1,733° F), the slenderness ratio (l/r) for the bars being in the general range 20 to 150. One group of tests with cast-iron specimens is included for comparative purposes. Since the compressive strength of a material is influenced greatly by the dimensions of the specimen, a range in shape and proportion was introduced. This extended from specimens of relatively thin material and unsymmetrical section that failed by local buckling to fully symmetrical sections proportioned to act as homogeneous units under load application. For the latter type of specimen the main element affecting strength at a given temperature was found to be the slenderness ratio.

Two general methods of testing are included—one in which the specimen is heated to a given temperature and then loaded to failure, and the other in which the load is maintained constant and the temperature increased until failure occurs. The latter is designed to simulate the condition to which building columns are subjected when exposed to fire. While the effects of duration of load and temperature were determined from the standpoint of fire exposure from fires in buildings, the results should not be taken as applicable to the design of columns to be subjected to load and high temperatures for longer periods.

With relatively short-length shaped specimens, an ultimate strength appreciably higher than the yield point was obtained at room temperatures, except with the light-angle and pressed-steel sections. Round bars of smaller diameter than ¾ in. (45 l/r) did not develop a higher strength than the yield point. For structural steel tested at temperatures of 250° C (482° F) or higher, and for cast iron at all temperatures, no well-defined yield point or yield region was developed. A strength higher than that obtained at room temperatures was developed at temperatures near 250° C (482° F) in all tests except those with the light-angle, pressed-steel, and cast-iron sections, and with round bars of smaller diameter than ¾ in. (45 l/r). In the tests with cast-iron specimens, which were all of low slenderness ratio (23 l/r), the strength developed was a little higher than any obtained with structural steel for the same temperature and slenderness ratio.

Agreement in point of ultimate strength at given temperatures and slenderness ratios was found with results from two series of fire tests of

building columns. Techn. Pap. BS 15 (1921) T184 and BS J. Research **10**, 737 (1933) RP563.

The ultimate strength of round bars tested at constant temperature in the range 450 to 600° C (842 to 1,112° F) and of columns failing under constant load at temperatures between 500 and 635° C (932 to 1,175° F) is given within plus or minus 15 percent by the formula

$$P/A = 10{,}000\left(\frac{1{,}870}{T}\right)^{3}\left(\frac{r}{l}\right)^{1/9}$$

where P/A is the average stress in pounds per square inch, T is the temperature at failure in degrees centigrade, and l/r is the slenderness ratio.

The extent of axial deformation at failure was found to be largely a function of slenderness ratio and shape, the symmetrical specimens having the lower slenderness ratios developing the greatest deformations.

The results of expansion determinations taken in conjunction with the stress-strain relations defined for given temperatures indicated that if building members are restrained by the surrounding construction and heated to a higher temperature, stresses induced by the restraint may become higher than those due to the supported load.

EFFECT OF COLD-ROLLING ON THE INDENTATION HARDNESS OF COPPER

The hardness of many metals can be increased by cold-working, i. e., by progressive drawing, rolling, or hammering of the metal without annealing. Copper is generally classed as one of the work-hardenable metals, but there have been reports of irregularities and of reversals in the hardness-reduction relation during severe cold-working of copper. The Bureau has completed an investigation in which tough-pitch electrolytic copper, oxygen-free copper, and large single crystals of copper were subjected to severe cold-rolling. It was found that the indentation hardness increased with successive reductions, rapidly at first, until a maximum hardness was reached. The maximum hardness was maintained thereafter during further reduction until the hardness determinations became unreliable owing to the thinness of the specimens. Irregularities in the hardness-reduction relation were encountered only in the case of very thin specimens. Similar results were obtained for the change in tensile strength with increasing reduction. The change in mechanical properties, as a result of severe cold-rolling, was not appreciably affected by the initial thickness of the specimen, the oxygen content of the copper, the change from polycrystalline to single-crystal material, or the orientation of the single-crystal specimens with respect to the plane of deformation. The reported irregularities in the hardness-reduction relation were not confirmed. The complete account of this investigation is published as RP742 in the Journal of Research for November.

PREPARATION OF PURE GALLIUM

Pure gallium was prepared from crude material containing 5 percent of indium, 0.1 percent of lead, and smaller amounts of silver, tin, and zinc, at the National Bureau of Standards to provide metal for use as an alloying element in the cadmium-vapor arc lamp and for researches in spectroscopy. In order that the method might be of general use for the purification of gallium, separations were devised to remove any impurities that are likely to be found in crude gallium. As a result a method was developed in which the principal operations consist in (1) preparing a hydrochloric acid solution of the metal and extracting the gallium, molybdenum, gold, iron, and thallium, together with small amounts of other elements, with ether; (2) precipitating antimony, arsenic, bismuth, cadmium, copper, germanium, gold, mercury, silver, and tin, and most of the lead, molybdenum, and rhenium with hydrogen sulphide in an acid solution of the ether extract; (3) precipitating iron and thallium with sodium hydroxide and filtering; (4) depositing the gallium electrolytically from the alkaline filtrate; and (5) eliminating the remaining impurities by fractional crystallization of the metal. Indium and lead are the most persistent impurities, but the last traces can be removed by fractional crystallization.

Gallium (at least 99.999 percent pure), containing only very faint traces of iron, lead, and calcium and having a melting point of 29.780° C±0.005° C, was prepared. A complete account of this work appears as RP734 in the November number of the Journal of Research.

FREEZING POINT OF GALLIUM

Gallium is a metal having rather unusual properties. It is the only metal besides mercury which can exist as a liquid at room temperatures, and

as a consequence has been used in filling liquid-in-glass thermometers. Fused silica-glass thermometers filled with gallium are usable up to 981° C (1,800° F).

The temperature at which gallium changes from a liquid to a solid (or vice versa) is in the neighborhood of 29° C (85° F). Gallium is like water in that it expands on freezing. Only two other pure metals, bismuth and antimony, are known to possess this property. The familiar effects produced by the expansion of water on freezing do not occur with gallium, however, because gallium possesses the property of under-cooling, i. e., the liquid metal can be cooled well below its freezing point without solidifying. Gallium in silica-glass thermometers may be immersed in freezing water without danger of the metal solidifying.

A considerable quantity of this comparatively rare metal in a state of high purity was recently prepared at the Bureau (see preceding item) and an accurate determination of its freezing point has been made, as described in RP735 in the Journal of Research for November. The properties described above, together with a low thermal conductivity, made this determination a rather tedious one. The low thermal conductivity made it difficult to obtain a uniform temperature throughout the metal, and expansion on freezing caused breakage of the glass tubes in which the metal was contained. When the liquid gallium is cooled slightly below its freezing point and solidification started by dropping in some of the solid metal, the temperature suddenly jumps up to its freezing point.

The freezing or melting point was determined as 29.78° C (85.60° F) by observing the temperature at which the liquid and solid metal remained together without change.

DETERMINATION OF SILVER IN VERY DILUTE SOLUTIONS

Recent interest in the oligodynamic properties of silver—that is, its bactericidal powers—has brought about the demand for an accurate analytical method for determining silver in very dilute solutions. Concentration of silver of the order of 0.02 to 0.20 milligram per liter are concerned.

The research associates on engineering uses of silver at the Bureau have devoted some attention to this problem. Feigl's rhodanine test, which has been shown by Feigl and others to be accurate for quantitative estimation of silver in concentrations as low as 1 part in 5,000,000, has been selected as a promising method. The accuracy of this method in extremely dilute solutions was found to be very seriously affected by the adsorption of silver on the walls of glass vessels used in the analytical procedure. The amount of adsorption on glass may be considerably modified by the type of glass used or by certain treatments of the glass surface prior to use in the analytical procedure. This adsorption appears to be far less serious if vessels of fused quartz are used. With proper regard for these adsorption phenomena it appears, from preliminary experiments, that the rhodanine colorimetric method for silver will be applicable to concentrations of the order of those encountered in the bactericidal uses of this metal.

GOLD-CHROMIUM RESISTANCE ALLOYS

The unit of electrical resistance is maintained in national standardizing laboratories by means of wire-wound standards. The national laboratories are interested in improving the stability of these standards, either by improved methods of construction or by the development of more stable resistance alloys.

Besides stability, resistance alloys should have low temperature coefficients of resistance and low thermoelectric powers against copper in order that they may be readily measured to a high precision. It has been found that the addition of about 2 percent of chromium to gold will give an alloy the resistance of which is nearly independent of temperature over at least the interval 20° to 30° C. Tests recently made at the Bureau show gold-chromium alloys also to be exceptionally stable in electrical resistance, although their thermoelectric powers against copper are three or four times as large as that of manganin. The method of preparation and the properties of some of these gold-chromium alloys are described in RP737 in the November number of the Journal of Research.

TEMPERATURE-CONTROL BOX FOR STANDARD CELLS

A semiportable temperature control box has been constructed at the Bureau to keep a small group of saturated standard cells at a constant temperature, for use in cases where the customary unsaturated cell is not good enough. It consists of an outer aluminum case, the temperature of

which is automatically controlled by a conventional mercury-in-glass regulator and an inner aluminum case thermally insulated from the outer one. The inner case contains the standard cell. Details of construction are given in the November number of the Journal of Research (RP739). Four cells mounted in such a device and maintained at 33° C showing only such changes in emf as were exhibited by similar cells continuously maintained at 28° C. in an oil bath.

The complete box measures about 8 by 13 by 10 inches, weighs 22 pounds, and operates without attention when connected to a 110-volt ac circuit.

RESISTIVITY OF SULPHURIC-ACID SOLUTIONS

Operation of lead-acid storage batteries at low temperatures is often required on automobiles, airplanes, and pilot balloons. Dependability of such batteries is of vital importance, but it is well known that the performance of batteries is adversely affected when they are subjected to low temperatures. The physical properties of the electrolyte (solution of sulphuric acid) determine in large part the behavior of the battery. These properties include freezing points, change in density with temperature, electrochemical equivalents, viscosity, and electrical resistivity.

Until recently no data on the viscosity and resistivity of sulphuric acid at low temperatures have been available. Last year the Bureau published the results of its measurements on viscosity of solutions ranging in concentration from 15 to 45 percent sulphuric acid at temperatures between +30° C (+86° F) and −50° C (−58° F). Publication of a companion paper (RP738 in the November Journal of Research) is now announced, giving the resistivity of such solutions between +30° C (+86°F) and −40° C (−40° F).

In the course of the measurements it was found that the composition of solutions having minimum resistivity changes from about 31 percent at 30° C (86° F) to about 26.5 percent as the temperature is lowered to the freezing point. An empirical relationship between resistivity, kinematic viscosity, and the absolute temperature was found.

DETERMINATION OF SULPHUR AND SULPHATE IN WOOL

Methods for the quantitative determination of sulphur and sulphate in wool are described in RP731 in the November number of the Journal of Research. Sulphur is present in wool as an essential constituent of the wool substance and is considered by many to be an indication of the quality of the wool with regard to fineness, state of chemical deterioration, and damage due to such common factors as exposure to light and weather.

To determine the total sulphur in wool the specimen is ignited by means of an electric current in oxygen under pressure. The gases evolved contain the sulphur in an oxidized form and are absorbed in ammonium carbonate solution from which the sulphur is precipitated and determined gravimetrically as barium sulphate, which is weighed and the amount of sulphur calculated.

Wool may absorb and hold sulphur to which it is exposed in commercial processes, such as stoving, carbonizing, and dyeing. This affects many of its properties; for example, the ability of the wool to absorb and hold moisture. It is important to know the amount of sulphur in wool when changes in the properties of wool are to be followed or controlled.

To determine the amount of sulphate, the wool to be tested is dissolved in hydrochloric acid and the sulphate precipitated without preparatory oxidation. The sulphur normally present in the wool does not interfere with the determination of sulphur in the form of sulphate. It is estimated that with these methods either form of sulphur can be determined within 2 percent of the amount present.

IMPROVED EQUIPMENT FOR STUDYING AGING OF RUBBER

Improved equipment for studying the aging of rubber has recently been installed at the Bureau. The equipment is designed to maintain rubber test samples at a temperature of 160° F in circulating fresh air for considerable periods of time. Formerly large ovens were used for the purpose, but these had the disadvantage that samples sometimes became contaminated by volatile products from adjacent samples of different composition, so the present apparatus was built with a number of small ovens, one for each sample. To facilitate temperature control these ovens are mounted in thermostatically-controlled water baths. Circulation of fresh air is secured by providing each with a small inlet and a chimney. The insulation and distribution of heating elements are such that it is possible to obtain sufficiently uniform temperatures without stirring either the water bath or the air in the ovens.

USE OF SWEETPOTATO STARCH IN PAPER INDUSTRY

According to the results of the Bureau's experiments, sweetpotato culls offer some possibility as a source of starch for sizing paper and for paper adhesive. The culls constitute a huge waste in the Southern States, and this Bureau and the Bureau of Chemistry and Soils are jointly endeavoring to find ways of making profitable use of them.

Cornstarch and cassava starch are used extensively in book papers to improve their printing quality. The Bureau made and tested wood-fiber book papers sized with these starches and with sweetpotato starch, and the latter compared very favorably with the others. The same kind of experiments are now being made with rag-fiber papers.

Dextrin made from cassava starch, which is imported, is used as an adhesive for postage stamps, envelops, labels, and many other paper products, and for this purpose it is superior to the present cornstarch dextrins, as it has better adhesive quality. Gum made from sweetpotato dextrin, however, was apparently equal to cassava gum in all respects.

It is planned to extend these semi-commercial tests to trials on a commercial scale as soon as sufficient sweetpotato starch is made available through production of it in a commercial plant now being built.

RELATION OF PAPER PROPERTIES TO REGISTER IN OFFSET LITHOGRAPHY

As described in RP730 in the November number of the Journal of Research, the Bureau has tested out its recipe for the manufacture of wood-fiber lithographic paper, which minimizes misregister of color prints, with very satisfactory results. Through previous study, in both laboratory and printing plant, of commercial papers used for this purpose, it was found that most of the misregister troubles were caused by lengthwise expansion or contraction of the paper between printings. Noting that the least lengthwise change occurred when a large proportion of the fibers lay in that direction, the investigators evolved a way of manufacturing paper of maximum quality in this respect. Papers, made in accordance with the newly developed practice by three cooperating manufacturers, were subjected to experimental printing at the United States Coast and Geodetic Survey and improved register was obtained.

Paper used in offset lithography must have the proper "grain" for successful color printing since from 2 to 20 or more colors must fit with exactness one on top of another. Poor fitting of the colors, termed "misregister" among printers, causes serious economic losses. In cooperation with the Lithographic Technical Foundation, the Bureau is bringing to light much needed information on how this paper should be made and is thus helping to combat these losses.

METHODS FOR CALCULATING THE VOLUMETRIC COMPOSITION OF FLUID MIXTURES

Two limiting conditions under which fluids are mixed were discussed in the March number of Physics by S. H. Ingberg, chief of the Bureau's fire-resistance section. Under the first condition the volume and pressure of the fluid are maintained at initial values by providing means for free efflux, while the other involves retention of all original and added quantities during the mixing operation. The latter condition is possibly more often present and the methods of computing the volume of components are more obvious. No methods corresponding to the former condition have heretofore been available, and those applicable for the latter have been used instead, sometimes with considerable error in results.

The mathematical treatment developed applies in general to mixtures of fluids in a container or enclosure maintained at constant pressure and volume, assuming perfect and instantaneous diffusion, free efflux, and no reaction within the mixture. For comparison the equations for mixture within a container from which no efflux takes place are given, and also a treatment applicable to intermediate conditions. Specific applications are developed pertaining to the use of inert gas, such as carbon dioxide, released either from the liquefied form or as one of the inert components of flue gas or engine exhaust gas, to prevent fire and explosion in connection with hazardous processing and storage. Either an inert atmosphere is maintained continuously within the enclosure or a quantity of gas is stored ready for release by manual or automatic means in case fire occurs or a condition develops conducive to fire or explosion. The mathematical treatment applies for the latter condition and to requirements for subsequent ventilation of the spaces thus deluged to reduce the toxic-gas content and

raise the oxygen content to limits making such spaces safe for entry.

The development aided by curve sheets enables ready computation of the components of fluid mixtures and comparisons for differences in obtaining conditions. Thus, if one volume of fully inert gas is added to air under conditions permitting free efflux, its concentration in the mixture will be 0.63 and the oxygen ratio will be reduced to 0.078. If no efflux takes place during the admission the concentration of inert gas will be 0.50 and the oxygen ratio 0.105. If one volume of inert gas containing 5 percent oxygen is admitted, the oxygen concentration will be reduced to 0.108 for the condition of free efflux and to 0.13 if no gas is allowed to escape.

In the general case of dilution also, the condition of free efflux will accomplish a desired result with the least volume of diluent. While the difference for the two assumptions is not large for additions of less than one-half of the original quantity present, for two volumes of diluent the remaining concentration with free efflux will be about one-half of what would result if the components are contained during the operation. On the assumption of free efflux, 4.6 volumes of diluent are needed to dilute a component from an initial concentration of 0.02 to 0.0002, as may be required for a toxic gas such as carbon monoxide before a space can be entered. On the assumption of no efflux, 99 volumes of air applied as a diluent would be needed to achieve the same result.

Considering that the influx tends to impel a portion of the contents of the container ahead of it, the efflux is likely to contain less of the component being added than its average concentration at the given instant for the space as a whole. Hence for conditions of free or partial efflux, the average concentration of the added component may accordingly be greater than as computed according to the development, which assumes complete and instantaneous diffusion. The relative position of inlets and outlets for mixture operations with given fluids can generally be arranged so that this condition will be assured.

FRACTIONATION OF THE ISOTOPES OF HYDROGEN AND OF OXYGEN

The electrolytic fractionation of the isotopes of hydrogen and of oxygen under the conditions prevailing in a commercial hydrogen-oxygen electrolyzer has been followed at the Bureau by means of measurements of density. Upon electrolysis of ordinary water the change in density at the beginning is caused as much by the fractionation of oxygen as of hydrogen. After an amount of water approximately equal to 10 times the volume of a commercial alkaline cell has been electrolyzed, a steady state is closely approached in which no further isotopic fractionation occurs. In this state the gases evolved have the isotopic composition existing in the water added to the cell, and the residual water left in the cell is 60 parts per million heavier than ordinary water. Of this 60 parts per million, 28 are contributed by the heavy isotope of hydrogen and the remainder by heavy oxygen. A more complete description of these observations is published as RP729 in the November number of the Journal of Research.

POLYMERIZATION OF OLEFINS

The purpose of the investigation described in RP740 in the November number of the Journal of Research was to determine the structure of the diisoamylenes prepared by the action of sulphuric acid on methylisopropylcarbinol in order to add further to our knowledge of the mechanism of polymerization of simple olefins to complex oils and resins. It was found that the action of 75-percent sulphuric acid on methylisopropylcarbinol at 80° C results in the formation of two isomeric decenes, namely, 3, 4, 5, 5-tetramethylhexene-2 and 3, 5, 5-trimethylheptene-2. The theory proposed by Whitmore for the polymerization of olefins does not explain the formation of the above products without the postulation of a complicated rearrangement which his experimental work on the dehydration of alcohols has shown to be unlikely. A theory is proposed for the polymerization of olefins based on the apparent activation of the olefin molecule, causing it to behave as two fragments which then add to the double bond of another olefin molecule. This behavior is analogous to the addition of RMgX to a carbonyl group $=C=O$ to yield $=C(R)-OMgX$. This mechanism satisfactorily explains the formation of the observed diisoamylenes and the di- and tri-isobutylenes. When it is considered that there are 2,281 isomers with the empirical formula $C_{32}H_{34}$, the value of a simple mechanism which permits the prediction of the products of polymerization of simple olefins becomes evident. From a consideration of the relative calorie strengths of bonds,

it is predicted that during the addition of the activated olefin molecule to another olefin molecule a C-C bond would be broken more readily than a C-H bond, which is approximately 22,000 calories stronger. Thus the products to be expected differ markedly in many cases from those predicted on the basis of a labile hydrogen mechanism.

SECOND SPECTRUM OF HAFNIUM

Hafnium was first isolated and recognized as a new chemical element in 1923. An extensive description of its emission spectra comprising 1,500 lines was made at the Bureau in 1928, and shortly thereafter regularities were discovered in its first two spectra. It was found, however, that the analyses of spectral structures could not be carried to a satisfactory conclusion without first obtaining additional information about the spectral lines. With new and purer material, the arc and spark spectra have been remeasured, as described in the November number of the Journal of Research (RP732). Observations were extended both to shorter and to longer waves, so that the number of observed lines has been doubled, and, in addition, the behavior of 280 lines was studied in strong magnetic fields. With the aid of these new data, nearly 900 lines characteristic of singly ionized hafnium atoms have been interpreted. They arise from energy changes among the same type of valence electrons that are present in neutral lanthanum, and a remarkable resemblance between the two spectral, Hf II and La I, is seen. The existence of 14 so-called "rare-earth elements" between these two has been assumed to be due to the addition of 14 new electrons in an inner shell, and structural analyses of the emission spectra of lanthanum and hafnium confirm this hypothesis.

BACTERICIDAL EFFECTS OF X-RAYS

A note on the bactericidal effects of X-rays, based on measurements at the Bureau, appears as RP736 in the November number of the Journal of Research. The effects of X-rays in liquids are localized in columns of ionization along the path of each high-speed electron. It is assumed that biological effects are produced in these columns and experimental results of Wyckoff and Rivers on killing of colon bacilli by cathode rays and X-rays are expressed in terms of the effective collision area of the column and bacillus for killing. Cathode rays of 155 kv give an area of 0.68×10^{-10} cm^2. X-rays of wave lengths ranging from 0.55 A to 4 A give values between 0.72×10^{-10} and 2.6×10^{-10} cm^2. As the area of the bacillus is about 10^{-5} cm^2 passage of a column through the bacillus is in general not fatal. The column must pass through a particular point to cause death.

IONIZATION OF LIQUID CARBON DISULPHIDE BY X-RAYS

The ionization of carbon disulphide by X-rays was measured in a layer of liquid between flat aluminum disks with spacings of 1 and 0.3 mm. Fields up to 60 kv per cm were used and the current voltage curve rose continuously, but at a decreasing rate in this range. A plot of the reciprocal of the current against the reciprocal of the field is a straight line above 20 kv and can be extrapolated to infinite field. The form of the curve is consistent with the theory of columnar recombination, and the intercept at infinite field gives the rate of production of ions. Comparison with the radiation in roentgen units gives the ionization per unit volume in liquid carbon disulphide as 2,600 times that of air under standard conditions. The absorption is 1,910 times that of air and the energy of ionization about 0.75 times that of air. The shape of the curve and the columnar theory give the diameter of the columns as 5.8×10^{-6} cm and the density of the ionization in the columns as 2.3×10^{16} per cm^3. More complete details of these measurements will be found in RP733 in the Journal of Research for November.

MEETING OF OPTICAL SOCIETY OF AMERICA

The nineteenth annual meeting of the Optical Society of America was held at the Bureau on October 18 to 20, inclusive. In all, 26 papers were presented, including 4 by members of the Bureau's staff, as follows: *Surface color*, by D. B. Judd; *Aviation lighting research at the National Bureau of Standards*, by F. C. Breckenridge; *A convenient set of equations for computing third-order aberrations*, by I. C. Gardner; *A testing camera for photographic objectives*, by I. C. Gardner and F. A. Case; *A filter isolating 560 millimicrons*, by K. S. Gibson.

In addition to the papers presented at the regular sessions, two public lectures were given on the evening of October 19. The first was by Charles Bittinger, of Washington, and dealt with ultraviolet murals at the Frank-

lin Institute in Philadelphia. Mr. Bittinger explained the principles upon which the painting of these pictures depends and illustrated these principles by actually painting a picture with ultraviolet pigments.

In the second lecture Dr. F. G. Pease, of Mount Wilson Observatory, discussed the many problems involved in the design of large telescopes. A considerable part of the lecture was devoted to the new 200-inch telescope. The various types of mountings which have been considered were compared and their advantages and disadvantages explained. The new design of ribbed disk which has been developed by the Corning Glass Works and an improved lever system for supporting the mirror without flexure were also described. Another revolutionary development is the aluminizing of the mirror, which gives a far more durable reflecting surface than silver.

An exhibit of optical instruments was arranged by the Bureau in connection with the meeting, many of the exhibits being of such a character as to illustrate fundamental optical laws.

On the evening of October 18 the delegates were received by Capt. J. F. Hellweg and the staff of the United States Naval Observatory. The reception was followed by an inspection of the Observatory's equipment, which includes the famous 26-inch refractor and the new 40-inch reflector now in process of construction. The sending of time signals at 10 p. m. was an interesting feature.

The meeting was attended by 75 members and the lecture room was filled to capacity on October 19.

SIMPLIFICATION OF INDUSTRIAL TRUCK AND TRAILER TIRES APPROVED

The simplified practice recommendation for industrial truck and trailer tires, cured-on type, having been accorded the required degree of written approval by the industry, became effective November 1, 1934. This simplified schedule of sizes will be included in the current revision of Simplified Practice Recommendation R103–33, which applies to industrial truck and trailer tires of the pressed-on type and which become effective June 1, 1934. The new schedule for the cured-on type of tire was proposed and developed by the standing committee for Simplified Practice Recommendation R103–33 and includes the stock sizes of the cured-on type of solid tires for use on industrial trucks and trailers.

SIMPLIFICATION OF CUPOLA REFRACTORIES

Simplified Practice Recommendation R154–34, covering cupola refractories, is now available in printed form. This recommendation, proposed and formulated by the industry, contains a simplified list of sizes for cupola blocks, tap-out blocks, and slag-hole blocks.

There are five tables in the recommendation, as follows: Table 1, 9- by 6- by 4-inch cupola blocks; table 2, 9- by 4½- by 4-inch cupola blocks; table 3, two-hole tap-out blocks; table 4, one-hole tap-out blocks; and table 5, slag-hole blocks.

The recommendation represents a substantial reduction in the variety heretofore listed for regular stock purposes, especially for tap-out and slag-hole blocks.

The printed edition of the recommendation also contains a summary of the development of the project, the membership of the industry's standing committee, and a list of the associations and firms that have accepted the program. The publication contains five illustrations. Copies may be obtained from the Superintendent of Documents, Government Printing Office, Washington, D. C., at 5 cents apiece.

COMMERCIAL STANDARD FOR INTERCHANGEABLE GROUND-GLASS JOINTS, STOPCOCKS, AND STOPPERS

The purpose of Commercial Standard CS21–34, which has just been issued in printed form, is to provide dimensional requirements for obtaining, within practical limits, interchangeability in ground-glass joints, stopcocks, and stoppers for ordinary laboratory and industrial work.

This standard covers: (1) Interchangeable ground-glass joints from 3 mm to 65 mm for laboratory and industrial glassware; (2) interchangeable straight-bore, ground-glass stopcocks from 1 mm to 10 mm bore; (3) interchangeable ground-glass stoppers from 8 mm to 35 mm diameters for volumetric flasks, stoppered Erlenmeyer flasks, stoppered cylinders, separatory funnels, and iodine-determination flasks; and (4) interchangeable ground-glass stoppers from 12.5 mm to 40.3 mm diameters for reagent bottles.

The standard also covers material and construction requirements for standard master plug and ring gages with dimensional tolerances, the required fit of mating gages with reference to each other, and fit of the finished product in working gages. The

universal taper for all interchangeable ground-glass joints, stopcocks, and stoppers is 1 to 10, the specified length of the ground zone varying with the product. A complete set of master gages is maintained by the industry at the Bureau for reference.

The success of the commercial standard for interchangeable ground-glass joints, CS21–30, led the standing committee to extend the principle of interchangeability to stopcocks and stoppers. While the details of the specifications have been carefully formulated, they may be considered for a period of time as on trial and subject to revision to suit composite experience. The personnel of the standing committee is given in the printed pamphlet, together with a list of acceptors and a brief history of the project.

Interchangeable ground-glass joints, stopcocks, and stoppers are identified by the letters "ST" entwined as a symbol on each member, followed by the size number and trade mark of the manufacturer.

This publication is obtainable from the Superintendent of Documents, Government Printing Office, Washington, D. C., at 5 cents per copy.

LIST OF AERONAUTICAL PUBLICATIONS

The Bureau's mimeographed list of aeronautical publications, Letter Circular LC325, has just been revised and will be mailed on request to anyone having a real use for it. The majority of the reports of the Bureau's work in aeronautics have been published not in its own series of publications but in the Technical Reports and Technical Notes of the National Advisory Committee for Aeronautics and in scientific and technical journals. The present list is, therefore, the only one which includes all of the Bureau's many contributions in this field and it should prove valuable for reference purposes. The publications are grouped under six heads: Aerodynamics; aircraft materials and construction; aeronautic power plants; aircraft instruments; aids to air navigation; and miscellaneous.

NEW AND REVISED PUBLICATIONS ISSUED DURING OCTOBER 1934

Journal of Research [1]

Journal of Research of the National Bureau of Standards, vol. 13, no. 4, October 1934 (RP nos. 718 to 728, inclusive). Price 25 cents. Obtainable by subscription.

Research Papers [1]

[Reprints from the June and August Journal of Research]

RP689. Iodine number of wool: A method for determining the action of various chemical reagents on wool and other proteins; Milton Harris, Harvey A. Neville, and William C. Fritz. Price 5 cents.

RP699. Derivation of photometric standards for tungsten-filament lamps; H. T. Wensel, William F. Roeser, L. E. Barbrow, and F. R. Caldwell. Price 5 cents.

RP701. Heats of combustion and of formation of the normal aliphatic alcohols in the gaseous and liquid states, and the energies of their atomic linkages; Frederick D. Rossini. Price 5 cents.

RP703. Refractive index and dispersion of normal and heavy water; L. W. Tilton and J. K. Taylor. Price 5 cents.

RP705. The system: $PbO–SiO_2$; R. F. Geller, A. S. Creamer, and E. N. Bunting. Price 5 cents.

Simplified Practice Recommendations [1]

R152–34. Basic dimensions for cones for warp and knitting yarns and hole sizes for bobbins for filling cop winders. Price 5 cents.

R154–34. Cupola refractories. Price 5 cents.

Commercial Standards [1]

CS21–34 (2d ed.). Interchangeable ground-glass joints, stopcocks, and stoppers. Price 5 cents.

CS48–34. Domestic burners for Pennsylvania anthracite (underfeed type). Price 5 cents.

Technical News Bulletin [1]

Technical News Bulletin No. 210, October 1934. Price 5 cents. Obtainable by subscription.

[1] Send orders for publications under this heading only to the Superintendent of Documents, Government Printing Office, Washington, D. C. Subscription to Technical News Bulletin, 50 cents per year; Journal of Research, $2.50 per year (United States and its possessions, Canada, Cuba, Mexico, Newfoundland, and Republic of Panama); other countries, 70 cents and $3.25, respectively.

LETTER CIRCULARS [2]

It is the intent of the Bureau to distribute single copies of these Letter Circulars on request only to those parties having special interest in the individual letter circular. Economy necessitates limitation in the number of copies issued. It is not the intent to supply parties with a copy of each Letter Circular issued during the month. Letter Circulars are necessarily of a temporary nature designed to answer numerous inquiries on a given subject. Requests should be addressed to the National Bureau of Standards.

LC325. (Revised to Oct. 23, 1934.) Aeronautical publications by members of the staff of the National Bureau of Standards.

LC425. Sound absorption coefficients of the more common materials. (Supersedes LC359.)

LC426. Publications on the platinum metals.

LC427. Losses of gasoline in storage and handling.

LC428. House-plan services. (A list of organizations issuing house plans, floor plans, and suggestions for small-house design.)

OUTSIDE PUBLICATIONS [2]

Hall, E. L., and George, W. D., *Precision condenser calibration at radio frequencies*, Electronics (330 West Forty-second Street, New York, N. Y.), 7, 318 (October 1934).

Scott, G. N., *Application of a few statistical principles to corrosion problems*, Oil and Gas Journal (Tulsa, Okla.), 33, 74 (Oct. 25, 1934).

Marvin, C. F., Jr., *Observations of flame in an engine*, J. Soc. Auto. Eng. (Society of Automotive Engineers, 29 West Thirty-ninth Street, New York, N. Y.), 35, 391 (November 1934).

Allen, H. H., Rodgers, G. C., and Brooks, D. B., *Ice formation in aircraft-engine carburetors*, J. Soc. Auto. Eng. (Society of Automotive Engineers, 29 West Thirty-ninth Street, New York, N. Y.), 35, 417 (November 1934).

Drake, N. L., Kline, G. M., and Rose, W. G., *The diamylenes produced from methylisopropylcarbinol by sulphuric acid*, J. Am. Chem. Soc. (Mills Building, Washington, D. C.), 56, 2076 (October 1934).

Schoonover, I. C., *Silver equipment in chemical plants*, Chem. & Metal. Eng. (330 West Forty-second Street, New York, N. Y.), 41, 545 (October 1934).

Bogue, R. H., Lerch, W., and Taylor, W. C., *Influence of composition on volume constancy and salt resistance of portland-cement pastes*, Portland Cement Association Fellowship (National Bureau of Standards, Washington, D. C.), Paper no. 28 (October 1934).

[2] These publications are not obtainable from the Government unless otherwise noted. Requests should be sent direct to publishers.

U. S. GOVERNMENT PRINTING OFFICE: 1934